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Evidence for molecular N₂ bubble formation in a (Ga,Fe)N magnetic semiconductor

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Fe-doped GaN semiconductors are of interest for combining the properties of semiconductors and magnetic materials [1]. Depending on the growth temperature used, Fe can either be distributed homogeneously in the GaN host lattice or it can accumulate in the form of Fe-N nanocrystals. As a result of the small size of the nanocrystals and the sensitivity of Fe-doped GaN to specimen preparation for electron microscopy, the formation and physical properties of Fe-N nanocrystals in GaN are not yet fully understood.

The (Ga,Fe)N samples examined below were grown by metalorganic chemical vapour deposition [1] and studied in cross-sectional geometry using several transmission electron microscopy (TEM) techniques. Great care was taken during TEM specimen preparation to minimize Ar ion-beam induced artefacts. Fe-N nanocrystal formation was observed in samples that had been deposited at temperatures higher than 850°C. Interestingly, most of the Fe-N nanocrystals were found to be associated with closely adjacent void-like features in both TEM and scanning TEM (STEM) images, as shown in Figure 1(a). Here, we use aberration-corrected STEM and electron energy-loss spectroscopy (EELS) to show that these features are bubbles filled with molecular N₂. In order to interpret our experimental results, we calculate the N K spectrum for GaN using self-consistent real-space multiple-scattering calculations using FEFF 9.05, which allows to include the experimental conditions.

A dedicated STEM EELS measurement was performed across a single nanocrystal (shown in Fig. 1 (b)) embedded in the GaN host. A 100 kV acceleration voltage and a distributed-dose acquisition routine [2] was used to either minimize or control electron beam induced damage during the experiment. Figure 2 (a) shows selected N K edge spectra recorded from the nanocrystal, the adjacent N₂-containing region and the GaN host. The N K edge shows a typical three-peaked structure between 400 and 407 eV. Figure 2 (b) shows the experimental spectrum acquired from the bubble alongside with the experimental spectrum of N₂ [3] and simulation of a GaN spectrum.

Figure 3 shows the result of an experiment that provides direct evidence for the presence of N₂ gas by using the electron beam to puncture the bubble adjacent to the Fe-N nanocrystal and to release the gas into the microscope. We used a static sub-Angstrom beam with a current of about 350 pA to make a hole in the specimen exactly at the position of the bubble, while recording EELS spectra every 40 seconds. The intensity of the characteristic first peak of the N K edge at 401 eV was observed to decrease suddenly when the gas was released. The quantitative determination of the pressure of the N₂ gas in the bubble from the recorded EELS spectra is in progress.

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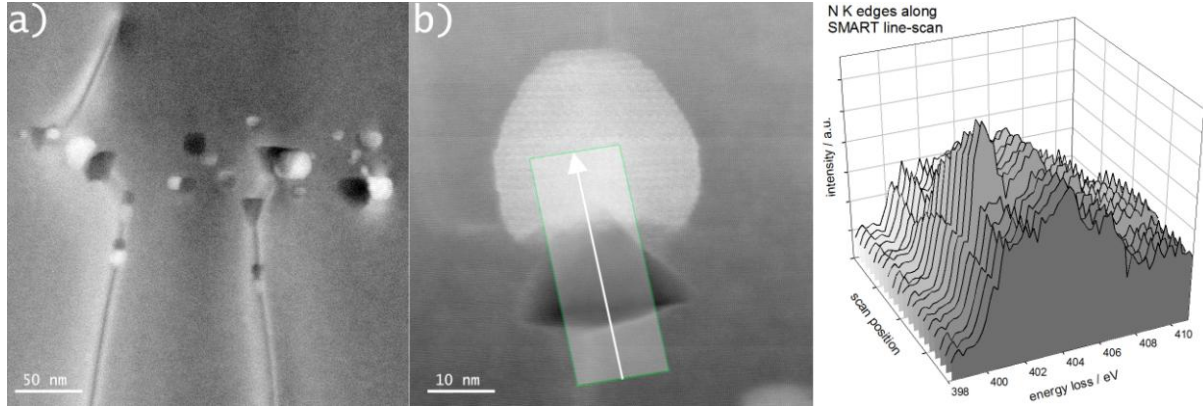


Figure 1. (a) HAADF STEM image of Fe-N nanocrystals and N_2 bubble in GaN. The nanocrystals appear with bright contrast. (b) Higher magnification image and averaged line-scan EELS measurement acquired from a single nanocrystal-bubble pair from the area indicated by the box. The EELS spectra show systematic changes in the N K edge fine structure.

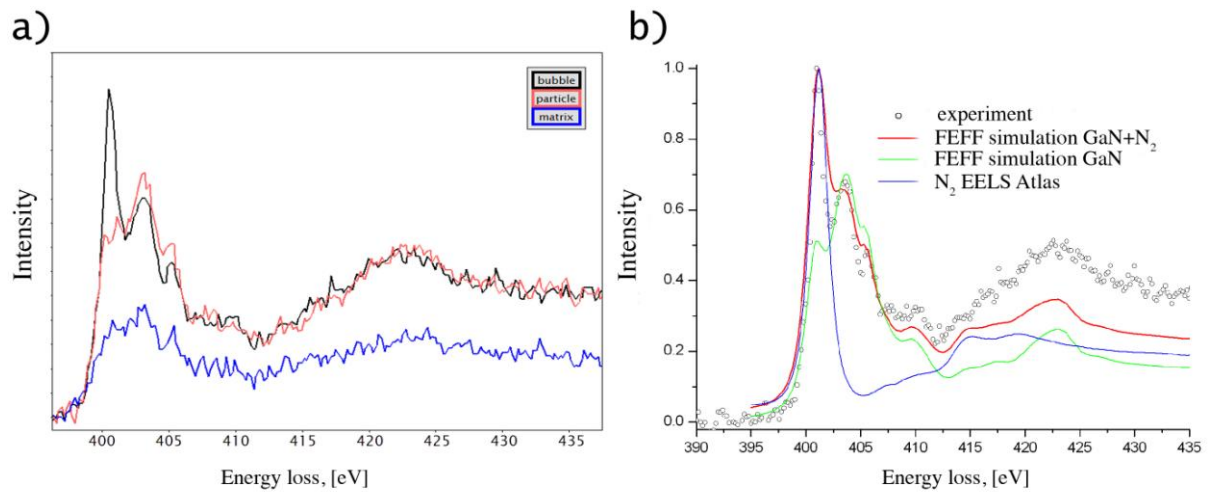


Figure 2. (a) Background-subtracted N K edge spectra measured from the Fe-N nanocrystal, N_2 bubble and GaN host lattice shown in Fig. 1. (b) Experimental spectra acquired from the bubble alongside an experimental measurement from N_2 from the EELS Atlas, and simulated spectra calculated for GaN.

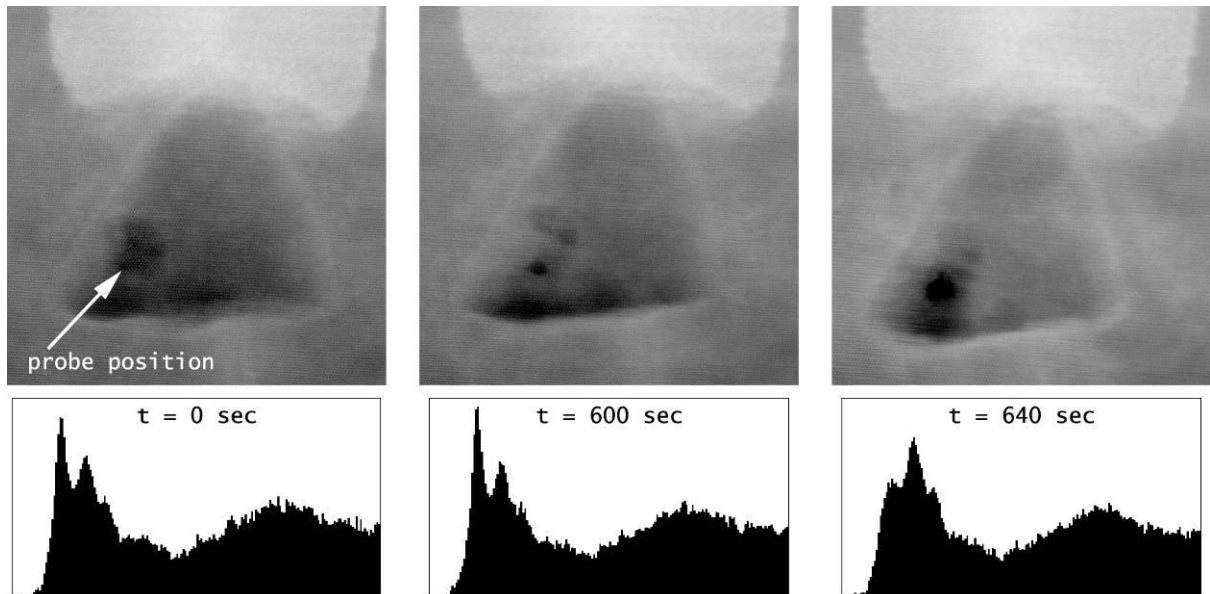


Figure 3. Time series of HAADF STEM images and N K edge measurements recorded while drilling a hole through the GaN using a stationary, focused electron probe. After 10 minutes a hole connects the bubble to the microscope vacuum and the N_2 gas is released. The intensity of the first peak in the spectrum is then reduced significantly.